

# REPORT DOCUMENTATION PAGE

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FROM: PROI (TI) (STINFO)

10 November 1999

SUBJECT: Authorization for Release of Technical Information, Control Number: AFRL-PR-ED-TP-1999-0215  
Veselenak, J., "Successful R&D Leveraging using T2 Mechanisms: Dual Use Polyhedral Oligomeric  
Silsequioxane (POSS) Nanotechnology"  
DoD Technology Transfer Integrated Planning Team Meeting (Statement A)

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Leilani Richardson

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(661) 275-5015

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ABSTRACT CLEARED 12 AUGUST 1999

Technical Abstract for DoD Technology Transfer Integrated Planning Team Meeting in Lake Tahoe, NV, Nov 16-18, 1999

AFRL-PR-ED-TP-FY99-062

Title: Military and Commercial Benefits of AFRL POSS Technology: Leveraging R&D Investments under Technology Transfer [This will be a slide presentation only.]

200-400 word abstract required by Army TTO, 12 Aug 99:

*In an attempt to meet the U.S. Air Force's demand for a new generation of lighter weight, higher performance polymeric materials, the U.S. Air Force Office of Scientific Research and the U.S. Air Force Research Laboratory Propulsion Directorate have, for the past six years, pursued the development of new chemical feedstock technologies based on Polyhedral Oligomeric Silsesquioxanes (POSS).*

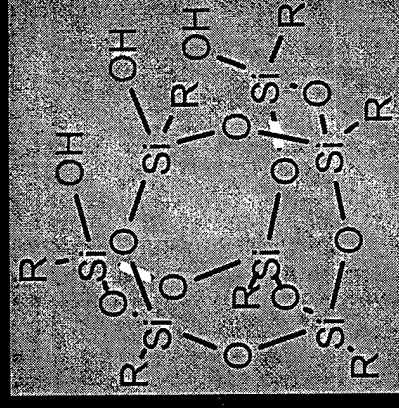
*Within six years this investment has paid off with the development and large-scale production of the first new polymer feedstocks in the past forty years. POSS technology is also the only hybrid and nanostructured, silicon-based chemical feedstock technology developed to date. Because of its chemical nature (an inorganic core with organic side arms), POSS technology is easily incorporated into common plastics via copolymerization or blending and hence requires little or no alteration to existing manufacturing processes. POSS additives radically upgrade the thermal and physical properties of most plastics.*

*The Hybrid Polymer Team is composed of highly motivated technology champions from not only the Air Force, but industry and academia as well. The Directorate has formed relatively seamless strategic alliances with the University of Dayton Research Institute, Hybrid Plastics, LLC, University of California at Irvine, Michigan State University, and the University of Michigan. As a result, the Air Force in-house team has been able to bring in over a hundred thousand dollars per year of non-Air Force money from small and medium sized chemical companies through use of the Cooperative Research and Development Agreement mechanism. In fact, their fiscal year 2000 commercial reimbursement may exceed \$250,000. Thus, the directorate's core competency has been sustained even though the number of Air Force assigned personnel has declined in response to significant budget cuts to Air Force Propulsion R&D.*

*POSS partnerships have paid off in several respects. First, they have leveraged Air Force funds (6.1, New World Vistas, and 6.2) and DoD (Dual Use Science & Technology Program) funds with other government (the Commerce Department's Advanced Technology Program) and industry investment to help incorporate the technology into dual use applications. Second, the partnerships' developmental work has resulted in promising potential applications, including lower erosion rocket motor insulation; plastic rocket engine ducting; long duration, supersonic jet canopies; nanostructured lubricants; and atomic oxygen and ultraviolet resistant coatings. This network of partnerships, made possible under multiple Technology Transfer mechanisms, serves as a model for successful public-private R&D collaboration.*

# Successful R&D Leveraging using T2 Mechanisms: Dual Use Polyhedral Oligomeric Silsesquioxane (POSS) Nanotechnology

Jeff Veselenak  
Manager, Technology Outreach Group  
Propulsion Directorate  
Air Force Research Laboratory

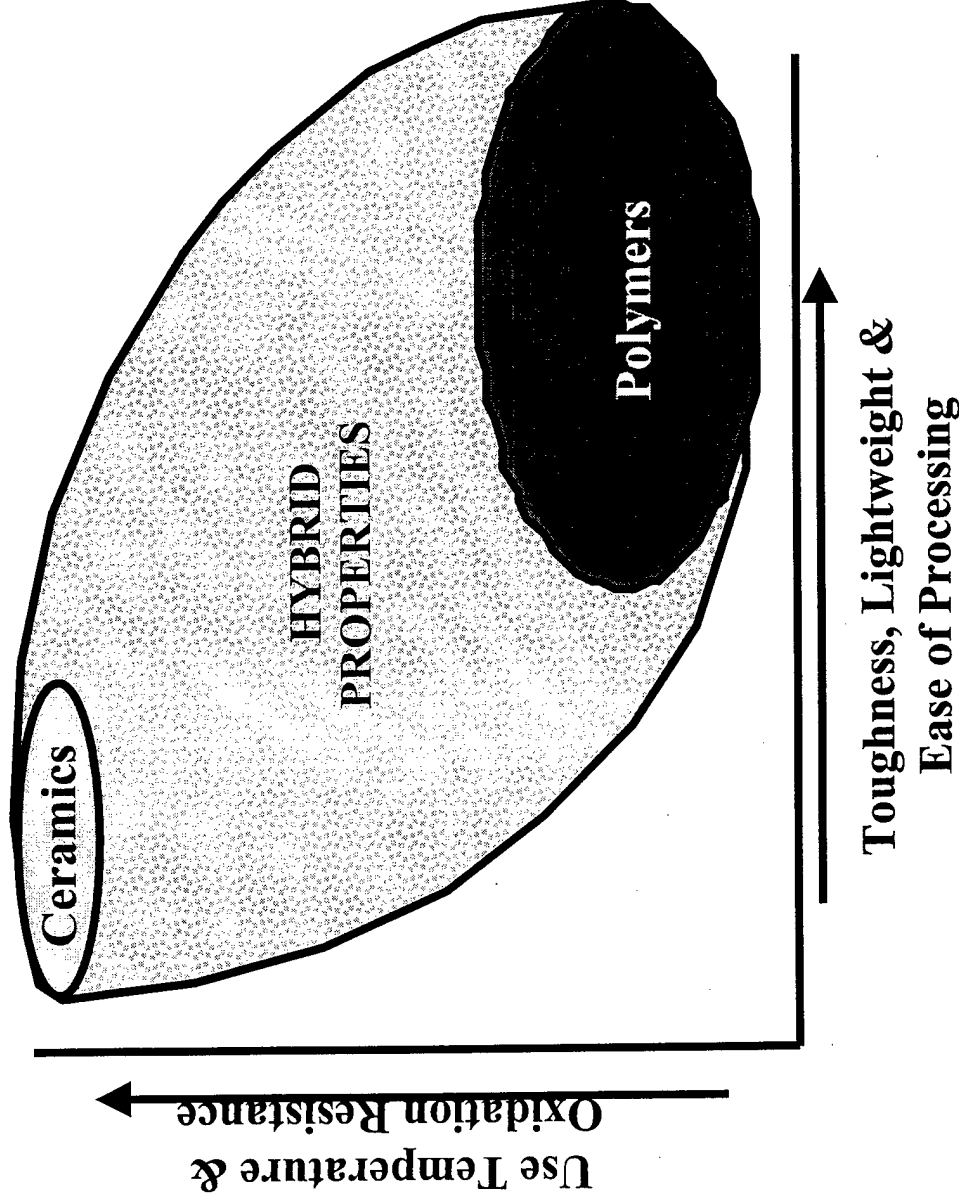


*for the*

DoD Technology Transfer Integrated Planning Team  
Incline Village, Nevada  
18 Nov 1999

## Propulsion (Air Force) Technology is Limited by Material Properties

Goal: Develop High Performance Polymers that REDEFINE material properties



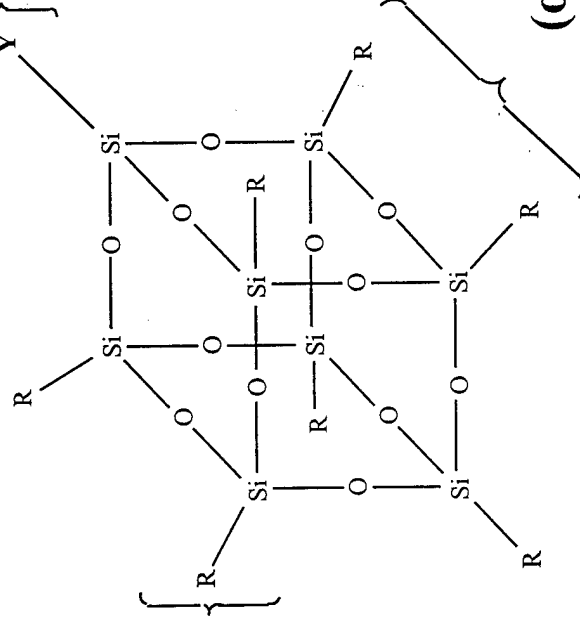
• Hybrid plastics can bridge the barrier between ceramics and polymers

# Anatomy of a POSS Molecule

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Nonreactive organic  
(R) groups for  
solubilization and  
compatibilization.

One or more reactive groups  
for polymerization or grafting.



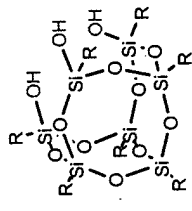
Thermally and chemically  
robust hybrid  
(organic-inorganic) framework.

Precise macromeric three-dimensional  
structure for molecular level reinforcement  
of polymer segments and coils.

# Property Enhancements via POSS

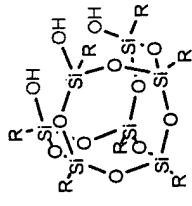
## Observed in POSS-Copolymers and Blends

increased $T_g$	increased $T_{dec}$	enhanced blend miscibility
reduced flammability	extended temperature range	oxidation resistance
reduced heat evolution	increased oxygen permeability	altered mechanicals
lower density	lower thermal conductivity	reduced viscosity
disposal as silica	thermoplastic or curable	



# Events Facilitated by DoD T2

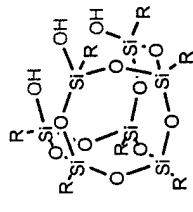
- 1996: Assignment of novel POSS structure and processing methods (patents) to UDRI
- 1996: Execution of a conduit (third party client) CRADA concept with UDRI--materials synthesis for reimbursement
- 1997: Polymer Working Group received AFOSR Star Team Award
- 1997: Significant increase of commercial interest in AFRL's POSS technology
- 1997: Inclusion of commercial funding into POSS R&D



# Events Facilitated by DoD T2

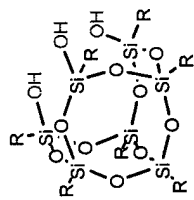
- 1998: Spin out of a commercial source (Hybrid Plastics, LLC (HP)) for bulk POSS monomer production
- 1998: Award of \$2M NIST ATP grant to HP
- 1998: Execution of a conduit CRADA between AFRL/PR and HP
- 1998: Tiger Team Assessment recommends WTN analysis
- 1999: WTN completes commercialization report
- 1999: Polymer Working Group and HP Team nominated for Gen Yates and FLC Awards





# T2 Mechanisms Employed

- CRADAs - Hybrid Plastics, UDRI
- SBIR Contract - Maxdem (Polymeric Ducting and Housing)
- DUS&T TIA - Wright Materials Research (aircraft canopies and radomes)
- PIA - WTN (commercialization of POSS, supplying applied research contacts)
- MOA/MOU - JPL/NASA: the movement towards funding for space experiments



# POTENTIAL MARKETS

- Rocket Propulsion Applications
- Air-Breathing Propulsion Applications
- Fire Resistant Materials
- Wire Insulation
- Contact Lenses
- Sporting Goods
- Blow Molded Films (Trash/Storage Bags)
- High Temperature epoxies and Resins
- Compounded Rubber
- Electronic Packaging
- Optical Plastics
- Dental Composites
- Structural Plastics
- R&D Chemicals

# POSS Technology Development Timeline

## Families of POSS-addition polymers

## Families of POSS-condensation polymers

POSS  
catalysis  
UCI

4 POSS-Polymer  
Architectures verified

POSS reagent  
scale-up method  
developed 0.5 kg +

International  
interest

"Targeted"  
resin  
development

POSS  
triol  
GE

POSS monomer  
polymerization  
verified

POSS reagent  
process  
improvement

POMS  
monomers  
developed

"Synthetic /  
processing"  
advances

## Technical Advances

1965 1989 1991 1992 1993 1994 1995 1996 1997 1998+

## Milestones

POSS nanocluster  
concept funded by  
PL and AFOSR

1st POSS-monomer  
tree completed  
(catalog)

Non DoD  
industrial  
product  
collaborations

Commercialization

DoD  
product  
collaborations

1st patent  
issued

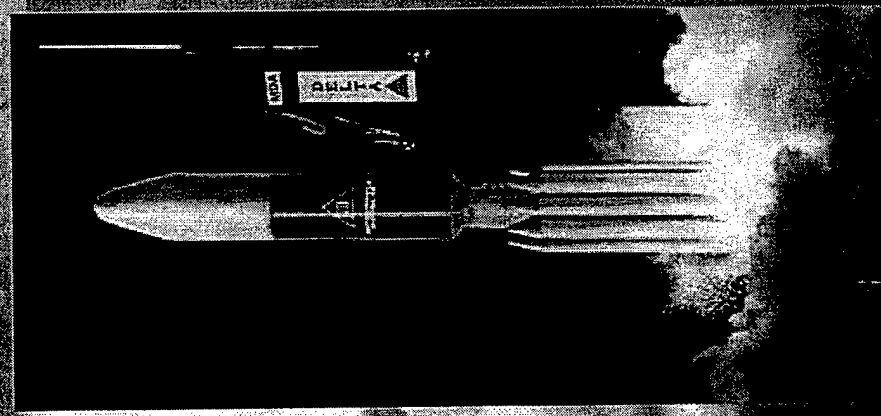
2nd patent  
issued

3rd patent  
issued

# High-Performance Nanotechnology Materials

## High-Performance Nanostructured Polymers

- Plastic tubing and ducting for liquid rockets engines
- High temperature case and motor insulation for solid rockets
- Space-resistant materials and coatings
- High-temperature canopies and hybrid lubricants

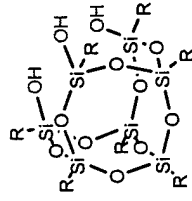


**High-Performance Nanotechnology Offers Versatility!**

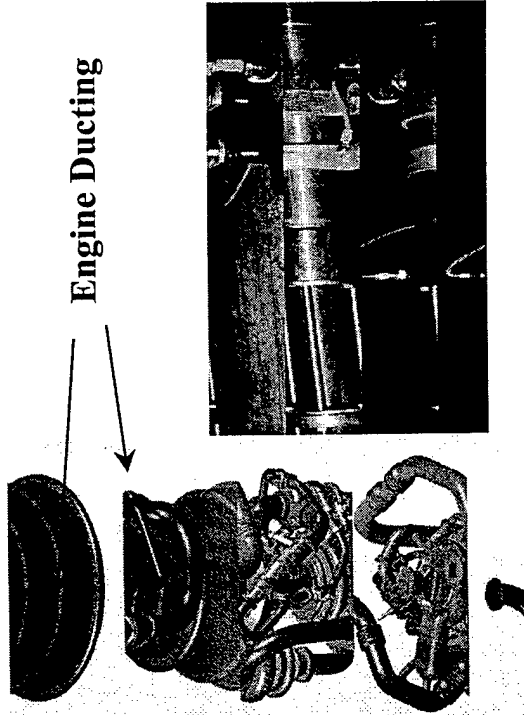
Take care  
that these  
pictures  
print  
properly.

# Plastics for Rockets

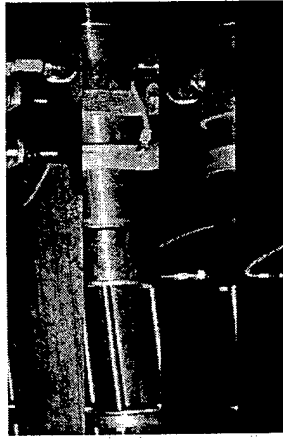
## Crucial to Reducing Weight and Cost



### Liquid Rocket Engines



*Polymer Tube/Case Hot Gas Burst Tester*

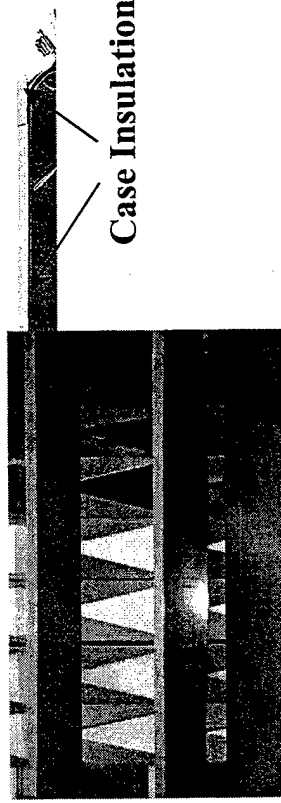
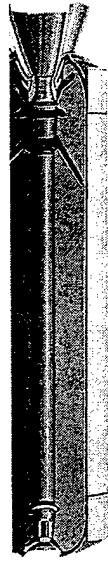


### Plastic Engine Ducting (SSME)

- 80% duct weight decrease
- 15% upper stage thrust-to-weight increase

3 candidates selected, SBIR, DUS&T

### Solid Rocket Motors



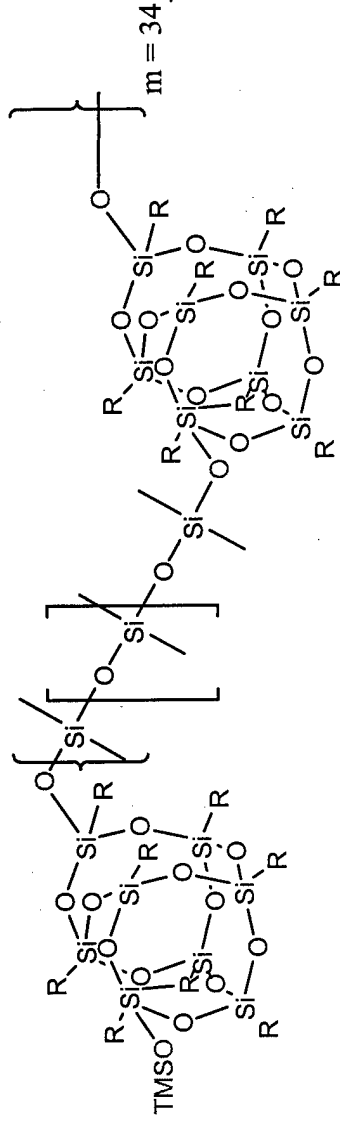
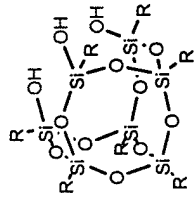
*Char Motor Polymer Insulation Samples*

### 50% Lower Erosion Insulation

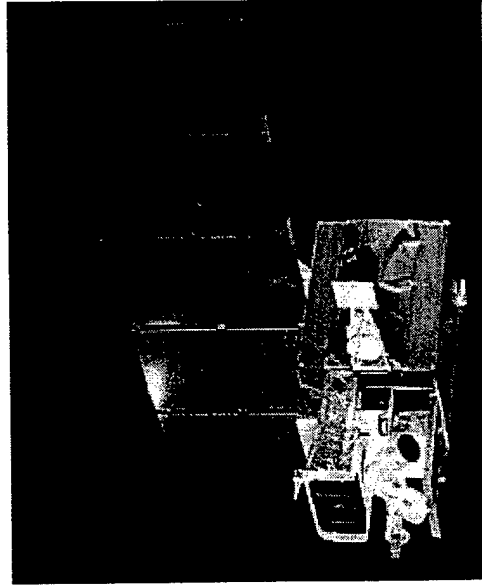
- Cuts Booster Insulation weight 44%
- Increases Booster Payload 7.4%

25% weight reduction & 6% density decrease  
Current testing with motor mfr. (30 lbs. POSS!)

# POSS Materials for Space Crucial to Reducing Weight and Cost



# POSS-PDMS copolymers



# Satellites & Space Systems

# POSS Nanocomposite Payoffs

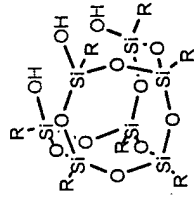
- **Maximum Space Resistance**
  - LEO, Atomic Oxygen (AO), VUV, Micro Impact
- **10% Lower Density**
- **High Modulus**
- **Resins for all Structural Applications**

### **Simulated 3 mo. AO/VUV Exposure**

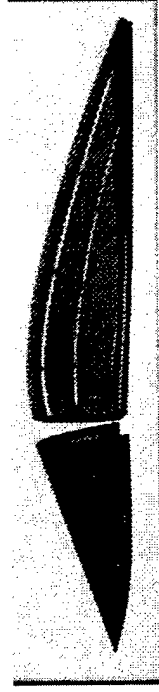
- **10x greater AO resistance than current state of art**
- **Even better AO/VUV resistance**
- **Annealing of surface microcracks!!!**
- **Space-Inflatables (AFRL/ML)**

**JPL collaboration, AO studies with Prof. Gar Hoflund, VUV with AFRL/ML**

# POSS Materials for Aerospace High Temperature & Lightweight



## Jet Canopies



### POSS-based Transparent Materials

- Mach 2.x speeds limited by plastic canopy (need increased HDT)
- Target Engagement Times can be reduced by increasing flight speed

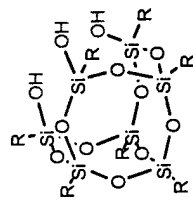
POSS-MMA increases use temp. by 150 °C  
POSS-polycarbonate currently being prepared  
Combining with nanocellular foam process  
*DUS&T with Wright Materials Research*

## Crucial Lubricant Applications



### Nanostructured Lubricants

- Current lubricants limited to 400 °F
- POSS based lubricants  $T_{dec} = 590$  °F
- Desire a fluid with working temperature range of -40° to 600° F (IHP/TET)



# POSS Applications Summary

## Addressing Propulsion Needs for High Performance Materials

- Lightweight, high-strength, high-temperature, & reduced cost
- Combine innovation with practicality
- Strong joint research effort with AFRL/ML-Materials Directorate
- Dual-use applications leveraged approach



# AFRL/PR-West Research Group (+ Future)

**Dr. Tim Haddad & Traudi Walker:**

Basic Research - POSS size and R group effects  
Applications - Jet Canopy, Radomes, Space

**Dr. Rusty Blanski & Justin Leland:**

Propulsion Applications - Lubes, Capacitors, Insulation  
Basic Research - POSS blends and additives

**Dr. Shawn Phillips & Dawn Hilton:**

Applications - LRE ducting tubing, Insulation  
Basic Research - high temp. polymers

**Pat Ruth:**

Basic Research - NWV Polymer Processing, blending  
Applications - capacitors, insulation

**Lt. Rene Gonzalez**

Space-Resistant Materials, High-temp. polymers

**Synthetic Post-Doc:**

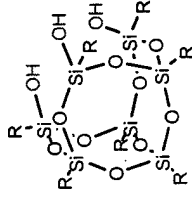
Commercial - Customer X  
Basic Research - R group effects  
Applications - Case Insulation

**2 Polymer Post-Docs + Assistant:**

Commercial - Hybrid Plastics ATP, Customer X  
Basic Research - R group effects  
Applications - Insulation, Tubing and Ducting

# AFRL Collaborations, Alliances, and Customers

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## POSS Monomers

Prof. Frank Feher\*: UC Irvine, POSS molecule synthesis

Prof. Rick Laine\*: U. of Mich., POSS molecule synthesis

Dr. Jim Spain: Tyndall AFB, POSS monomers via biocatalysis

Hybrid Plastics: CA, Supply of bulk POSS monomers for AF research

## POSS Polymers

Dr. Pat Mather: AFRL/ML, POSSnorbornyl, POSSpolyurethanes

Dr. Rich Vaia: AFRL/ML, POSSparmax, POSS/Clay Comp.

Prof. Ben Hsiao\*: New York U., POSSpolyurethanes

Prof. Andre Lee\*: Mich. State U., POSSepoxy polymers

Dr. Jeff Gilman\*: NIST, POSS ablative studies

Dr. Bill Wallace: NIST, Si-O-Si formation/opening

## POSS Computational

Dr. Mark Gordon: Iowa State U., POSS formation, POSS polymers

Dr. Barry Farmer: AFRL/ML, POSS polymers

## **Funding: AFRL, AFOSR, other**

\*Directly Funded by AFRL/PRSM

Update  
budget  
Change  
branch

# Collaborations, Alliances, and Customers

## Rocket Propulsion and Space Applications

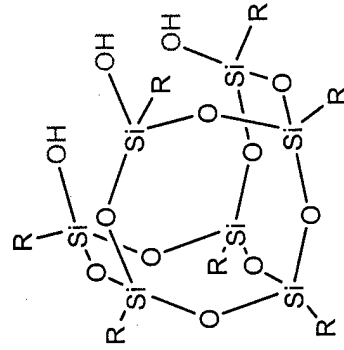
2 Customers + JPL

POSS Lubes

AFRL/PRSL/F

Fluorinated POSS

2 Customers



Hybrid Plastics

(40k FY99)

~~(180k FY00)~~

120

UDRI

(20k FY99)

(0K FY00)

Jet canopies/ Optical transparencies

Customer

Fire-safe Plastics

Customer/NIST/FAA

(60-150K FY00)

Commercial Sales

Aldrich/Gelest

WTN

Commercialization Support

Gas Separation

Customer

(60-120K FY00/01)

...and approximately 20 other proprietary customers.

# Departing Champions

AFRL Labor Drain Associated with POSS  
Monomer Production for Dual Use Applications  
Prior to Spin Out of Hybrid Plastics, LLC

- 1 On site contracted PhD. - Full Time
- 1 DR-II (GS-12 equiv.) - Full Time
- 1 On site contracted technician - Full Time
- 1 DR-III (GS-14 PhD. equiv.) - 1/4 Time

Over \$300K per year in salaries

# **Hybrid Plastics' Key Strategic Alliances**

## **Air Force Research Laboratory**

Propulsion Sciences and  
Advanced Concepts Directorate  
Edwards Air Force Base, CA

## **Accelerate Technology**

Small Business Development Center  
Irvine, CA

## **University of Dayton**

### **Research Institute**

Special Programs &  
Technology Commercialization  
Dayton, OH

## **University of California**

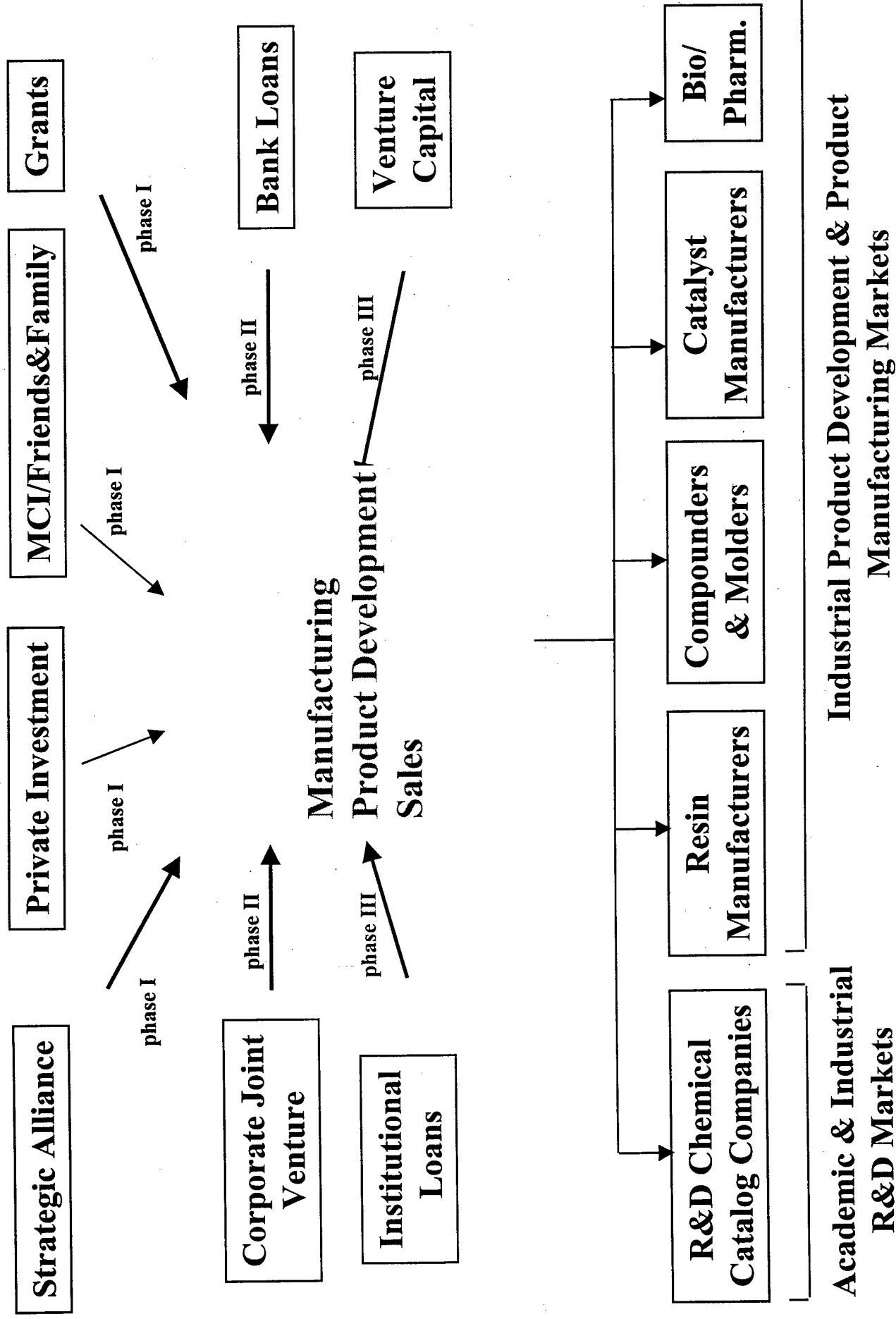
### **Irvine**

Department of Chemistry &  
Office of Technology Alliances  
Irvine, CA

## **National Institute of Standards and Technology**

Advanced Technology Program  
Gaithersburg, MD

# Hybrid Plastics' Business Spectrum



# **AFRL FY99-01 Polymer Research Goals**

## **Polymer Synthesis/Characterization Studies**

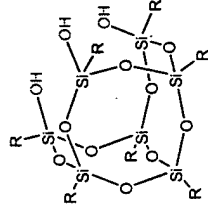
- **Reaction rates of POSS cages during copolymerization**
- **Type of POSS polymer formed (Bead vs. Pendant)**
- **Differences in size of POSS cages (Bead and Pendant)**
- **Varying non-reactive R groups (miscibility vs. agglomeration)**

## **Polymer Processing**

- **POSS miscibility for blends (R group effect)**
- **Processing environment on polymer structure - how does this effect physical and mechanical properties?**
- **Innovative processing techniques**
- **Blend miscibility of two different POSS polymers**

## **Polymer Studies Rely on Ability to Manipulate POSS Cages!!**

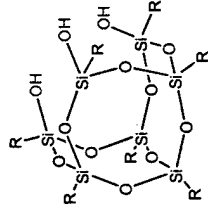
# The Future



## Push AFRL/PR and AF POSS Applications:

- Integrated High Payoff Rocket Propulsion Technology goals
- Synergy: Division (PRSL), Directorate (PRPE), AFRL (ML) DOD (Green Missile), NASA/JPL, DOC (NIST/FAA)
- Designed silicates (Clays, etc.,)
- Polymeric Cements (Geobond)
- Organometallic Polymers
- Functionally Graded Polymers (adhesion, property changes)
- Multi-Organometal Polymer Systems (smart systems)

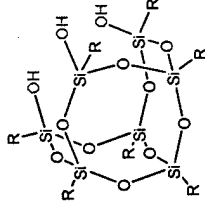




## What did AFRL gain?

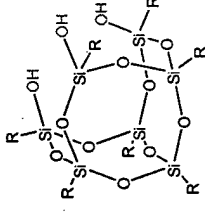
- Increased 6.1/6.2 R&D leveraging using a conduit CRADA
- External customer funds (approx.):
  - FY97 \$80K
  - FY98 50K
  - FY99 90K
  - FY00 est. 300K
- A commercial source of POSS monomer materials for research

# What did AFRL gain?



- Reduced, via commercial outsourcing, the labor drain associated with:
  - POSS monomer production - a technology we already developed and patented
  - POSS marketing
- Sustained a DoD core competency/center of excellence using “out of the box” approaches

# Lessons Learned from the Transfer of POSS Nanotechnology



- T2 champions are needed on both sides of the fence-a network of partnerships is best
- The champion S&Es should have an entrepreneurial interest in the advancement of the technology
- S&T funding cuts forced champions to think “out of the box” to advance the technology
- To gain trust of collaborators, the DoD must be diligent at protecting proprietary information-including its own
- The DoD must be aggressive with patenting its IP in hot fields